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**Patentanmeldung Nr. Patent application No. Demande de brevet n°**

02102448.4

Der Präsident des Europäischen Patentamts;  
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets  
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**R C van Dijk**





Anmeldung Nr:

Application no.: 02102448.4

Demande no:

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Anmelder/Applicant(s)/Demandeur(s):

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Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:

(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.

If no title is shown please refer to the description.

Si aucun titre n'est indiqué se referer à la description.)

Improved ink jet recording material

In Anspruch genommene Priorität(en) / Priority(ies) claimed /Priorité(s)  
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**[DESCRIPTION]**

## FIELD OF THE INVENTION

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The present invention relates to an ink jet recording material having an improved stabilization of the finished image against color fading due to light.

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## BACKGROUND OF THE INVENTION

In the majority of applications printing proceeds by pressure contact of an ink-loaden printing form with an ink-receiving material which is usually plain paper. The most frequently used impact printing technique is known as lithographic printing based on the selective acceptance of oleophilic ink on a suitable receptor. In recent times however so-called non-impact printing systems have replaced classical pressure-contact printing to some extent for specific applications. A survey is given e.g. in the book "Principles of Non Impact Printing" by Jerome L. Johnson (1986), Palatino Press, Irvine, CA 92715, USA.

Among non-impact printing techniques ink jet printing has become a popular technique because of its simplicity, convenience and low cost. Especially in those instances where a limited edition of the printed matter is needed ink jet printing has become a technology of choice. A recent survey on progress and trends in ink jet printing technology is given by Hue P. Le in *Journal of Imaging Science and Technology* Vol. 42 (1), Jan/Febr 1998.

In ink jet printing tiny drops of ink fluid are projected directly onto an ink receptor surface without physical contact between the printing device and the receptor. The printing device stores the printing data electronically and controls a mechanism for ejecting the drops image-wise. Printing is accomplished by moving the print head across the paper or vice versa. Early patents on ink jet printers include US 3,739,393, US 3,805,273 and US 3,891,121.

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The jetting of the ink droplets can be performed in several different ways. In a first type of process a continuous droplet stream is created by applying a pressure wave pattern. This process is known as continuous ink jet printing. In a first embodiment the droplet stream is divided into droplets that are electrostatically charged, deflected and recollected, and into droplets that remain uncharged, continue their way undeflected, and form the image. Alternatively, the charged deflected stream forms the image and the uncharged undeflected jet is recollected. In this variant of continuous ink jet printing several jets are deflected to a different degree and thus record the image (multideflection system). According to a second process the ink droplets can be created "on demand" ("DOD" or "drop on demand" method) whereby the printing device ejects the droplets only when they are used in imaging on a receiver thereby avoiding the complexity of drop charging, deflection hardware, and ink recollection. In drop-on-demand the ink droplet can be formed by means of a pressure wave created by a mechanical motion of a piezoelectric transducer (so-called "piezo method"), or by means of discrete thermal pushes (so-called "bubble jet" method, or "thermal jet" method).

Ink compositions for ink jet typically include following ingredients : dyes or pigments, water and/or organic solvents, humectants such as glycols, detergents, thickeners, polymeric binders, preservatives, etc.. It will be readily understood that the optimal composition of such an ink is dependent on the ink jetting method used and on the nature of the substrate to be printed. The ink compositions can be roughly divided in :

- water based ; the drying mechanism involves absorption, penetration and evaporation;
- oil based ; the drying involves absorption and penetration;
- solvent based ; the drying mechanism involves primarily evaporation;
- hot melt or phase change : the ink vehicle is liquid at the ejection temperature but solid at room temperature ; drying is replaced by solidification;
- UV-curable ; drying is replaced by polymerization.



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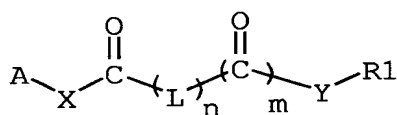
It is known that the ink-receiving layers in ink-jet recording elements must meet different stringent requirements :

- The ink-receiving layer should have a high ink absorbing capacity, so that the dots will not flow out and will not be expanded more than is necessary to obtain a high optical density.
  - The ink-receiving layer should have a high ink absorbing speed (short ink drying time) so that the ink droplets will not feather if smeared immediately after applying.
  - The ink dots that are applied to the ink-receiving layer should be substantially round in shape and smooth at their peripheries. The dot diameter must be constant and accurately controlled.
  - The receiving layer must be readily wetted so that there is no "puddling", i.e. coalescence of adjacent ink dots, and an earlier absorbed ink drop should not show any "bleeding", i.e. overlap with neighbouring or later placed dots.
  - Transparent ink-jet recording elements must have a low haze-value and be excellent in transmittance properties.
  - After being printed the image must have a good resistance regarding water-fastness, light-fastness, and good endurance under severe conditions of temperature and humidity.
  - The ink jet recording element may not show any curl or sticky behaviour if stacked before or after being printed.
  - The ink jet recording element must be able to move smoothly through different types of printers.
- All these properties are often in a relation of trade-off. It is difficult to satisfy them all at the same time.

A particular problem is the stability of the color densities of the finished color ink jet image when exposed to light for a longer period ("light-fastness"). As well-known by those skilled in the art the light fading of colorants is mainly due to an oxidative decomposition of the colorant catalyzed by light, in particular by the UV spectral part. Therefore, there is a permanent need of more effective compounds which stabilize the colorants of the ink jet image against fading by light.

## SUMMARY OF THE INVENTION

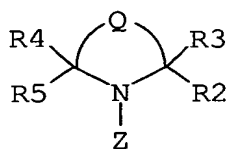
The above-mentioned advantageous effects are realised by providing an ink jet recording material comprising a support and at least one  
 5 ink receiving layer containing a binder and a compound according to following general formula (I) :



formula (I)

10

wherein A is represented by following formula :



15 wherein,

Q represents the necessary atoms to complete a five- or six-membered ring;

R2 to R5 independently represent a substituted or unsubstituted C1 to C6 aliphatic group;

20 Z is selected from the group consisting of hydrogen, a substituted or unsubstituted aliphatic group, an acyl group, an oxy radical, a hydroxyl group, an alkoxy group and an acyloxy group;

L represents a divalent linking group;

X and Y are independently selected from an oxygen and NR6, wherein

25 R6 is selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group; X is linked to A via one of the atoms of Q;

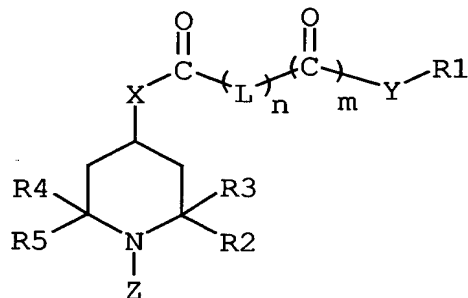
30 R1 represents a non-aromatic moiety comprising at least one hydroxyl

- 6 -

group;

n and m independently represent 1 or 0.

Preferably, the ring is a six-membered ring with X in 4-position  
 5 versus N, so that the general formula of the light-stabilizing  
 compound becomes :



formula (II)

10 wherein,

R2 to R5 independently represent a substituted or unsubstituted C1  
 to C6 aliphatic group;

Z is selected from the group consisting of hydrogen, a substituted  
 or unsubstituted aliphatic group, an acyl group, an oxy radical, a  
 15 hydroxyl group, an alkoxy group and an acyloxy group;

L represents a divalent linking group;

X and Y are independently selected from an oxygen and NR6, wherein  
 R6 is selected from the group consisting of hydrogen, a substituted  
 or unsubstituted, saturated or unsaturated aliphatic group, a  
 20 substituted or unsubstituted aromatic group, and a substituted or  
 unsubstituted heteroaromatic group;

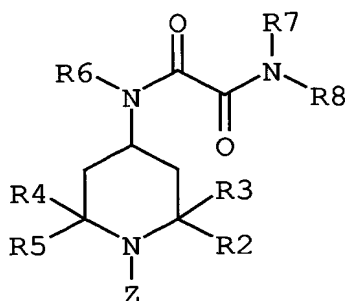
R1 represents a non-aromatic moiety comprising at least one hydroxyl  
 group;

n and m independently represent 1 or 0.

25

In a particularly preferred embodiment the light-stabilizing  
 compound is an oxalamide derivative according to following general  
 formula:

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formula (III)

wherein,

5 R2 to R5 independently represent a substituted or unsubstituted C1 to C6 aliphatic group;

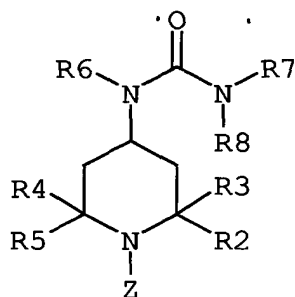
Z is selected from the group consisting of hydrogen, a substituted or unsubstituted aliphatic group, an acyl group, an oxy radical, a hydroxyl group, an alkoxy group and an acyloxy group;

10 R6 is selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group;

R7 and R8 are independently selected from the group consisting of  
 15 hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group, with the proviso that at least one of R7 and R8 represents a non-aromatic moiety comprising at least one hydroxyl group.

20

According to another particularly preferred embodiment the light-stabilizing compound is an urea derivative according to following general formula :



formula (IV)

5 wherein,

R2 to R5 independently represent a substituted or unsubstituted C1 to C6 aliphatic group;

Z is selected from the group consisting of hydrogen, a substituted or unsubstituted aliphatic group, an acyl group, an oxy radical, a hydroxyl group, an alkoxy group and an acyloxy group;

R6 is selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group;

15 R7 and R8 are independently selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group, with the proviso that at least one of R7 and R8 represents a non-aromatic moiety comprising at least one hydroxyl group.

Further advantages and embodiments of the present invention will become apparent from the following description.

## 25 DETAILED DESCRIPTION OF THE INVENTION

The different layers and particular ingredients of the ink recording medium according to the present invention will now be explained in detail.

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**- the support**

The support for use in the present invention can be chosen from paper type and polymeric type supports well-known from photographic technology. Paper types include plain paper, cast coated paper, polyethylene coated paper and polypropylene coated paper. Polymeric supports include cellulose acetate propionate or cellulose acetate butyrate, polyesters such as polyethylene terephthalate and polyethylene naphthalate, polyamides, polycarbonates, polyimides, polyolefins, poly(vinylacetals), polyvinyl chloride, polyethers and polysulfonamides. Other examples of useful high-quality polymeric supports for the present invention include opaque white polyesters and extrusion blends of polyethylene terephthalate and polypropylene. Polyester film supports and especially polyethylene terephthalate are preferred because of their excellent properties of dimensional stability. When such a polyester is used as the support material, a subbing layer may be employed to improve the bonding of the ink-receiving layer to the support. Useful subbing layers for this purpose are well known in the photographic art and include, for example, polymers of vinylidene chloride such as vinylidene chloride /acrylonitrile /acrylic acid terpolymers or vinylidene chloride /methyl acrylate /itaconic acid terpolymers.

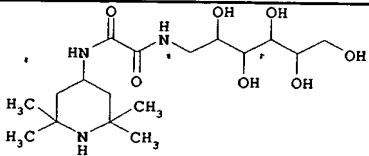
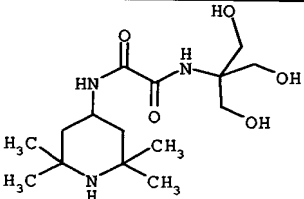
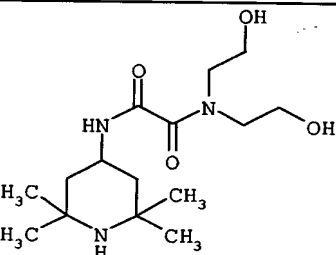
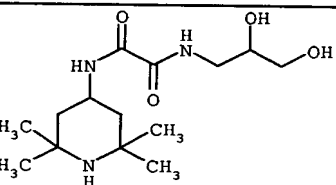
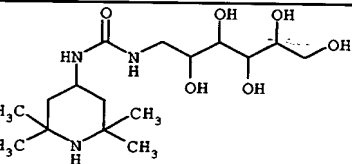
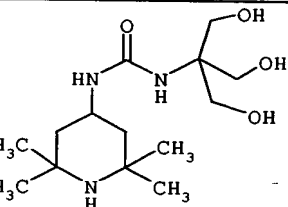
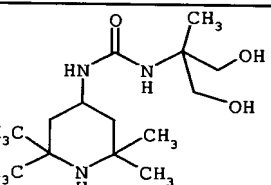
**- the ink receiving layer**

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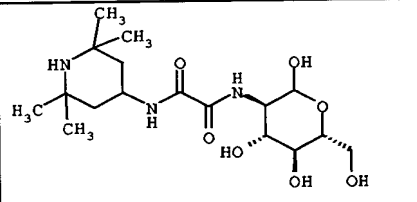
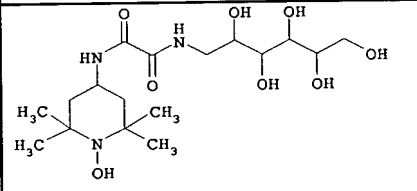
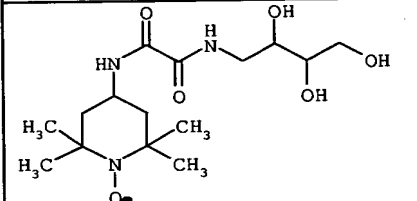
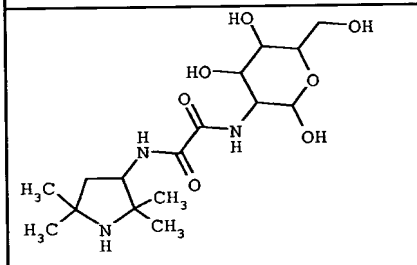
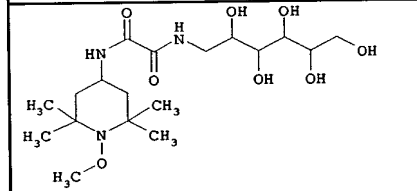
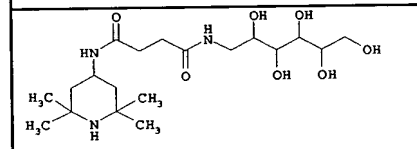
It is the gist of the present invention that the ink receiving layer contains, apart from a binder, a light-stabilizer according to general formula (I), preferably according to formula (II), and most preferably according to formula (III) or formula (IV).

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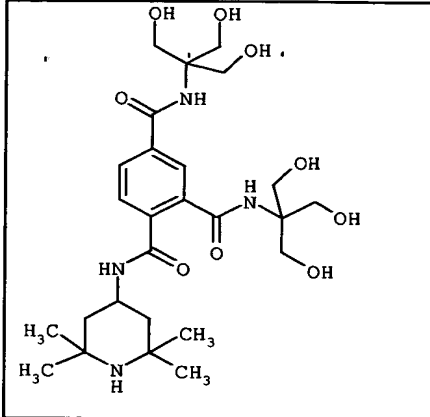
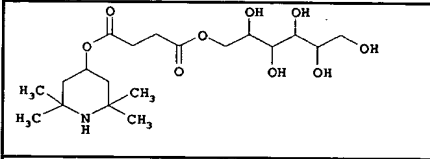
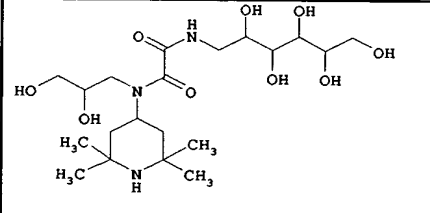
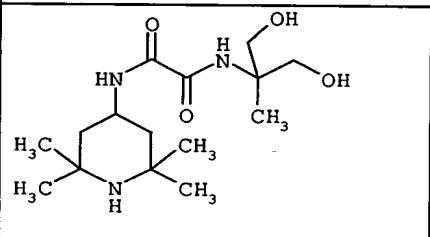
Typical examples of light-stabilizing compounds used in accordance with the present invention are included in following list without being limited thereto.

	Stabilizer 1
	Stabilizer 2
	Stabilizer 3
	Stabilizer 4
	Stabilizer 5
	Stabilizer 6
	Stabilizer 7

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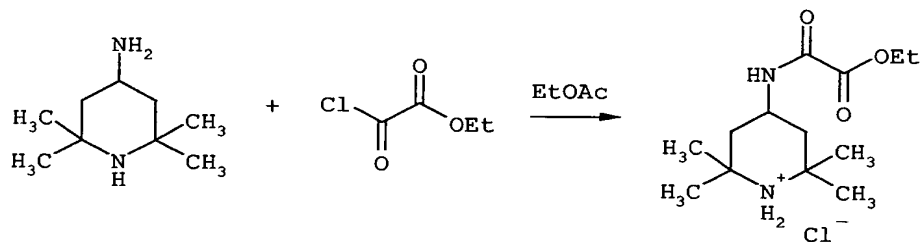
	Stabilizer 8
	Stabilizer 9
	Stabilizer 10
	Stabilizer 11
	Stabilizer 12
	Stabilizer 13



	Stabilizer 14
	Stabilizer 15
	Stabilizer 16
	Stabilizer 17

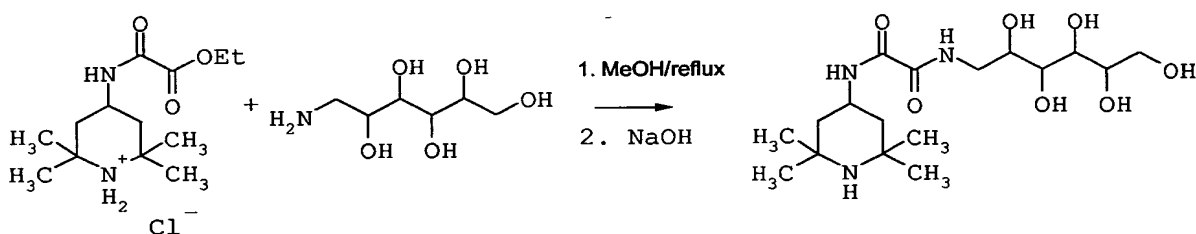
All compounds can be prepared according to well-known synthetic procedures. The synthesis of HALS-compounds used in accordance with the present invention is described more in detail below, to  
 5 illustrate the invention.

**The synthesis of stabilizer 1 :**



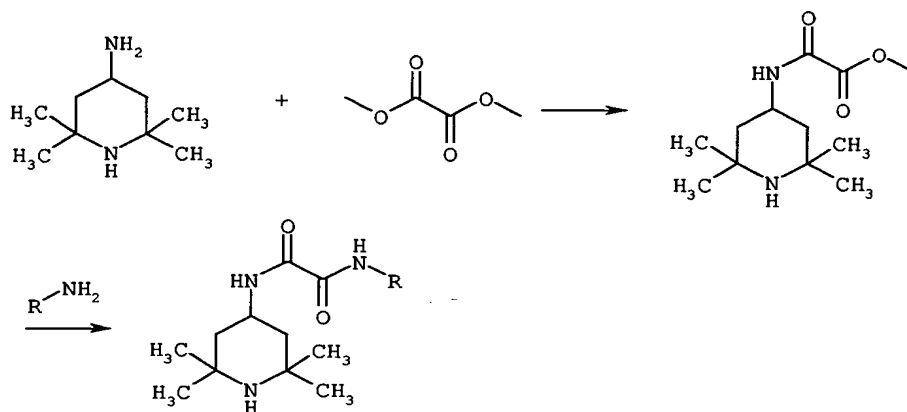
- 13 -

87.5 ml (0.5 mol) of 4-amino-2,2,6,6-tetramethyl-piperidine was dissolved in 390 ml of ethyl acetate. 56 ml (0.5 mol) of ethyl oxalyl chloride was added dropwise, while keeping the temperature at 40°C. The reaction product precipitated from the medium. Upon  
 5 complete addition, the reaction was allowed to continue for 2 hours at room temperature. The compound was isolated by filtration, washed twice with 50 ml of ethyl acetate and dried. 127 g (87%) of the oxalamide chlorohydrate was isolated.



32.2 g (0.11 mol) of the oxalyl amide chlorohydrate was dissolved in 275 ml of methanol. While stirring, 22 g (0.12 mol) of glucamine was added in 3 portions. The reaction mixture was refluxed for 7 hours. After cooling down to room temperature, the reaction mixture was  
 15 concentrated to 150 ml. 300 ml of ethyl acetate was added and an oily product precipitated. The oily residue was treated several times with methanol/ethyl acetate 1/1 and finally with 300 ml of methyl-tert.butyl ether. On treating with methyl-tert. butyl ether, the product solidified and was isolated by filtration. 34.5 g of a  
 20 crude chlorohydrate was isolated. Based on NP thin layer chromatography (eluent : CH<sub>2</sub>Cl<sub>2</sub>/MeOH/NH<sub>3</sub>(aq) : 70/24/6), it was judged that the HALS-oxalyl amide was insufficiently pure for evaluation. To remove the impurities, the chlorohydrate was converted into the free base with sodium hydroxide as follows. 34.5  
 25 g of the crude chlorohydrate was dissolved in 180 ml of water. 40 ml of 2N NaOH was added and the mixture was extracted 4 times with 180 ml of ethyl acetate. The aqueous fraction was isolated and water was removed under reduced pressure. The oily residue was treated with 180 ml of ethanol and the precipitated salts were removed by  
 30 filtration. Ethanol was removed under reduced pressure and finally 17.7 g of compound 1 was isolated.

**The synthesis of stabilizers 2, 4 and 17 :**



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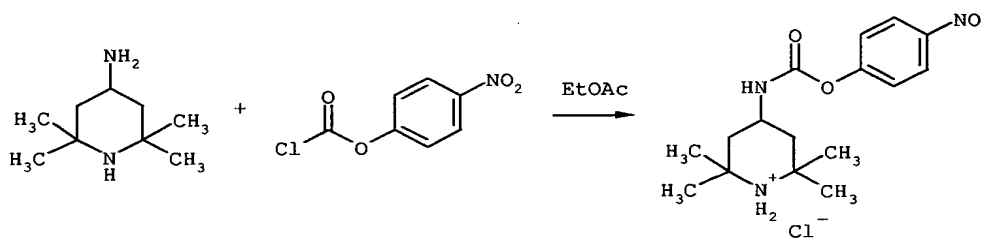
R-NH <sub>2</sub>	Stabilizer
	Stabilizer 2
	Stabilizer 17
	Stabilizer 4

A solution of 15.6 g (0.1 mol) of 4-amino-2,2,6,6-tetramethylpiperidine in 30 ml of methanol was added dropwise to a solution of 14.26 g (0.12 mol) of dimethyl oxalate in 70 ml of methanol, while keeping the temperature at room temperature. The reaction was allowed to continue at room temperature for 2 hours. After two hours, 0.1 mol of the appropriate amine was added and the reaction was allowed to continue at room temperature for 16 hours. The precipitated side products were removed by filtration and methanol was removed under reduced pressure. The different stabilizers were purified by reversed phase chromatography on

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Kromasil C18 100 A 10 $\mu$ m, using a gradient elution, starting with MeOH/water, buffered with ammonium acetate at pH=7.6 (0.2M), 1/9 and ending with MeOH/water, buffered with ammonium acetate at pH=7.6 (0.2M), 38/62, using a flow rate of 150 ml/min. 11 g (33 %) of stabilizer 2, 9.4 g (30 %) of stabilizer 17 and 13.2 g (44%) of stabilizer 4 were isolated, partially as acetate salt.

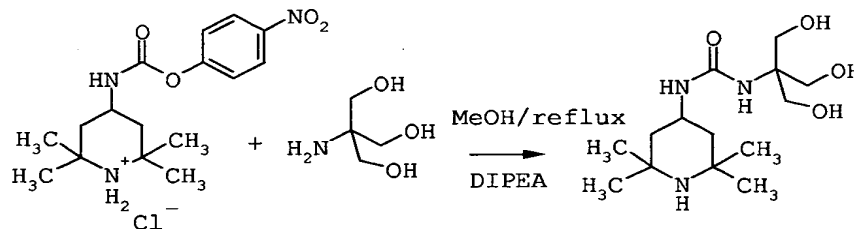
**The synthesis of stabilizer 6 :**



10

3.4 g (16.8 mmol) of 4-nitrophenyl chloroformate was dissolved in 40 ml of ethyl acetate. The reaction mixture was cooled to 0°C and a solution of 2.39 g (15.3 mmol) of 4-amino-2,2,6,6-tetramethylpiperidine in 40 ml of ethyl acetate was added over 5 minutes. Immediately, the chlorohydrate of the 4-nitrophenyl-carbamate started to precipitate as a white solid. The reaction was allowed to continue for 1 hour at room temperature. The precipitated chlorohydrate was isolated by filtration, washed with ethyl acetate and dried. 5.0 g (91%) of the carbamate was isolated. The compound was sufficiently pure to be used without further purification.

20



25

2 g (5.6 mmol) of the 4-nitrophenyl-carbamate was dissolved in 50 ml of methanol. 1.6 g (12.3 mmol) of DIPEA and 0.75 g (6.2 mmol) of tris(hydroxymethyl)aminomethane were added and the reaction mixture

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was refluxed for 3 hours. The solvent was removed under reduced pressure and stabilizer 6 was purified by reversed phase preparative column chromatography on Kromasil C18 100 A 10 $\mu$ m.

5 The ink receiving layer may consist of just one single layer, or alternatively it may be composed of two layers or even of multiple layers. A particular type of an extra top ink receiving layer may be designated as a so-called "gloss improving layer", meaning a layer which achieves a gloss of more than 30 at a 60 degrees angle. This  
10 gloss property can be achieved by the use of swellable polymers and/or (in)organic pigments with a particle size smaller than 500 nm.

In the case of double or multiple ink receiving layers the light-stabilizer may be incorporated in just one layer, or in several  
15 layers or in all layers. It may also be present in additional auxiliary layers if present, such as an anti-curl backing layer. The light-stabilizing compounds in connection with the present invention may also be incorporated in a typical ink jet ink, although they are more effective when present in ink jet media  
20 according to this invention.

The binder can be chosen from a list of compounds well-known in the art including hydroxyethyl cellulose; hydroxypropyl cellulose; hydroxyethylmethyl cellulose; hydroxypropyl methyl cellulose;  
25 hydroxybutylmethyl cellulose; methyl cellulose; sodium carboxymethyl cellulose; sodium carboxymethylhydroxyethyl cellulose; water soluble ethylhydroxyethyl cellulose; cellulose sulfate; polyvinyl alcohol; vinylalcohol copolymers; polyvinyl acetate; polyvinyl acetal; polyvinyl pyrrolidone; polyacrylamide; acrylamide/acrylic acid  
30 copolymer; polystyrene, styrene copolymers; acrylic or methacrylic polymers; styrene/acrylic copolymers; ethylene-vinylacetate copolymer; vinylmethyl ether/maleic acid copolymer; poly(2-acrylamido-2-methyl propane sulfonic acid); poly(diethylene triamine-co-adipic acid); polyvinyl pyridine; polyvinyl imidazole;  
35 polyethylene imine epichlorohydrin modified; polyethylene imine ethoxylated; polyethylene oxide; polyurethane; melamine resins;

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gelatin; carrageenan; dextran; gum arabic; casein; pectin; albumin; starch; collagen derivatives; collodion and agar-agar.

A preferred binder for the practice of the present invention is a polyvinylalcohol (PVA), a vinylalcohol copolymer or modified  
5 polyvinyl alcohol. Most preferably, the polyvinyl alcohol is a cationic type polyvinyl alcohol, such as the cationic polyvinyl alcohol grades from Kuraray, such as POVAL C506, POVAL C118, and from Nippon Goshei.

Mixtures of two or more binders can be used.

10

Apart from the binder and the light-stabilizer a pigment may be present in the ink receiving layer or in at least one of the ink-receiving layers, in the case of multiple layers.

The pigment used is preferably an inorganic pigment, which can be  
15 chosen from neutral, anionic and cationic pigment types. Useful pigments include e.g. silica, talc, clay, hydrotalcite, kaolin, diatomaceous earth, calcium carbonate, magnesium carbonate, basic magnesium carbonate, aluminosilicate, aluminum trihydroxide, aluminum oxide (alumina), titanium oxide, zinc oxide, barium  
20 sulfate, calcium sulfate, zinc sulfide, satin white, alumina hydrate such as boehmite, zirconium oxide or mixed oxides.

Preferably, the pigment is a cationic type pigment selected from alumina hydrates, aluminum oxides, aluminum hydroxides, aluminum silicates, and cationically modified silicas.

25 A preferred type of alumina hydrate is crystalline boehmite, or  $\gamma$ - $\text{AlO}(\text{OH})$ . Useful types of boehmite include, in powder form, DISPERAL, DISPERAL HP14 and DISPERAL 40 from Sasol, MARTOXIN VPP2000-2 and GL-3 from Martinswerk GmbH.; liquid boehmite alumina systems, e.g. DISPAL 23N4-20, DISPAL 14N-25, DISPERAL AL25 from Sasol. Patents on  
30 alumina hydrate include EP 500021, EP 634286, US 5,624,428, EP 742108, US 6,238,047, EP 622244, EP 810101, etc..

Useful cationic aluminum oxide (alumina) types include  $\alpha$ - $\text{Al}_2\text{O}_3$  types, such as NORTON E700, available from Saint-Gobain Ceramics & Plastics, Inc, and  $\gamma$ - $\text{Al}_2\text{O}_3$  types, such as ALUMINUM OXID C from  
35 Degussa; other aluminum oxide grades, such as BAIKALOX CR15 and CR30

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from Baikowski Chemie; DURALOX grades and MEDIALOX grades from Baikowski Chemie, BAIKALOX CR80, CR140, CR125, B105CR from Baikowski Chemie; CAB-O-SPERSE PG003 trademark from Cabot, CATALOX GRADES and CATAPAL GRADES from Sasol, such as PLURALOX HP14/150; colloidal

5 Al<sub>2</sub>O<sub>3</sub> types, such as ALUMINASOL 100; ALUMINASOL 200, ALUMINASOL 220, ALUMINASOL 300, and ALUMINASOL 520 trademarks from Nissan Chemical Industries or NALCO 8676 trademark from ONDEO Nalco.

Other useful cationic inorganic pigments include aluminum trihydroxides such as Bayerite, or  $\alpha$ -Al(OH)<sub>3</sub>, such as PLURAL BT,

10 available from Sasol, and Gibbsite, or  $\gamma$ -Al(OH)<sub>3</sub>, such as MARTINAL grades from Martinswerk GmbH, MARTIFIN grades, such as MARTIFIN OL104, MARTIFIN OL 107 and MARTIFIN OL111 from Martinswerk GmbH, MICRAL grades, such as MICRAL 1440, MICRAL 1500; MICRAL 632; MICRAL 855; MICRAL 916; MICRAL 932; MICRAL 932CM; MICRAL 9400 from JM Huber

15 company; HIGILITE grades, e.g. HIGILITE H42 or HIGILITE H43M from Showa Denka K.K..

Another useful type of cationic pigment is zirconium oxide such as NALCO OOS008 trademark of ONDEO Nalco, acetate stabilized ZrO<sub>2</sub>, ZR20/20, ZR50/20, ZR100/20 and ZRYS4 trademarks from Nyacol Nano

20 Technologies.

Useful mixed oxides are SIRAL grades from Sasol, colloidal metal oxides from Nalco such as Nalco 1056, Nalco TX10496, Nalco TX11678. Another preferred type of inorganic pigment is silica which can be used as such in its anionic form or after cationic modification.

25 Silica as pigment in ink receiving elements is disclosed in numerous old and recent patents, e.g. US 4,892,591, US 4,902,568, EP 373573, EP 423829, EP 487350, EP 493100, EP 514633, etc.. The silica can be chosen from different types, such as crystalline silica, amorphous silica, precipitated silica, fumed silica, silica gel, spherical and

30 non-spherical silica. The silica may contain minor amounts of metal oxides from the group Al, Zr, Ti. Useful types include AEROSIL OX50 (BET surface area  $50 \pm 15 \text{ m}^2/\text{g}$ , average primary particle size 40 nm, SiO<sub>2</sub> content > 99.8%, Al<sub>2</sub>O<sub>3</sub> content < 0.08%), AEROSIL MOX170 (BET

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surface area  $170 \text{ g/m}^2$ , average primary particle size  $15 \text{ nm}$ ,  $\text{SiO}_2$  content  $> 98.3\%$ ,  $\text{Al}_2\text{O}_3$  content  $0.3\text{--}1.3\%$ ), AEROSIL MOX80 (BET surface area  $80 \pm 20 \text{ g/m}^2$ , average primary particle size  $30 \text{ nm}$ ,  $\text{SiO}_2$  content  $> 98.3\%$ ,  $\text{Al}_2\text{O}_3$  content  $0.3\text{--}1.3\%$ ), or other hydrophilic AEROSIL

5 grades available from Degussa-Hüls AG, which may give aqueous dispersions with a small average particle size ( $<500 \text{ nm}$ ). Cationically modified silica can be prepared by following methods, without meaning to be limitative :

(1) subjecting silica to a surface treatment with an inorganic  
10 cationic compound such as particular metal oxides and oxyhydroxides, e.g. aluminum oxides, and alumina hydrates such as boehmite and pseudo-boehmite; a useful cationic inorganic compound to modify silica is pseudo-boehmite. Pseudo-boehmite is also called boehmite gel and is fine particulate alumina hydrate having a needle form.

15 The composition thereof is generally represented by  $\text{Al}_2\text{O}_3 \cdot 1.5\text{--}2 \text{ H}_2\text{O}$  and differs from that of crystalline boehmite;

(2) by subjecting silica to a surface treatment with an organic compound having both an amino group or quaternary ammonium group thereof or a quaternary phosphonium group, and a functional group  
20 having reactivity to a silanol group on the surface of silica, such as aminoalkoxysilane or aminoalkyl glycidyl ether or isopropanol amine;

(3) by polymerisation of a cationic or amino functional monomer in the presence of a silica.

25

In an alternative embodiment the pigment may be chosen from organic particles such as polystyrene, polymethyl methacrylate, silicones, melamine-formaldehyde condensation polymers, urea-formaldehyde condensation polymers, polyesters and polyamides. Mixtures of  
30 inorganic and organic pigments can be used. However, most preferably the pigment is an inorganic pigment.

Mixtures of two or more pigments may be used.



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For obtaining glossy ink receiving layers the particle size of the pigment should preferably be smaller than 500 nm. In order to obtain a porous glossy layer which can serve as an ink receiving layer for fast ink uptake the pigment/binder ratio should be at least 4. Only  
5 at these high ratios the binder is no longer able to fill up all pores and voids created by the pigments in the coating. To achieve a sufficient porosity of the coating for fast ink uptake the pore volume of these highly pigmented coatings should be higher than 0.1 ml/g of coated solids. This pore volume can be measured by gas  
10 adsorption (nitrogen) or by mercury diffusion.

Apart from the essential ingredients described above a cationic substance acting as mordant may be present in the ink receiving layer. Such substances increase the capacity of the layer for fixing  
15 and holding the dye of the ink droplets. A particularly suited compound is a poly(diallyldimethylammonium chloride) or, in short, a poly(DADMAC). These compounds are commercially available from several companies, e.g. Aldrich, Nalco, CIBA, Nitto Boseki Co., Clariant, BASF and EKA Chemicals.

20 Other useful cationic compounds include DADMAC copolymers such as copolymers with acrylamide, e.g. NALCO 1470 trade mark of ONDEO Nalco or PAS-J-81, trademark of Nitto Boseki Co., such as copolymers of DADMAC with acrylates, such as Nalco 8190, trademark of ONDEO Nalco; copolymers of DADMAC with SO<sub>2</sub>, such as PAS-A-1 or PAS-92, trademarks  
25 of Nitto Boseki Co., copolymer of DADMAC with maleic acid, e.g. PAS-410, trademark of Nitto Boseki Co., copolymer of DADMAC with diallyl(3-chloro-2-hydroxypropyl)amine hydrochloride, eg. PAS-880, trademark of Nitto Boseki Co., dimethylamine-epichlorohydrine copolymers, e.g. Nalco 7135, trademark of ONDEO Nalco or POLYFIX  
30 700, trade name of Showa High Polymer Co.; other POLYFIX grades which could be used are POLYFIX 601, POLYFIX 301, POLYFIX 301A, POLYFIX 250WS, and POLYFIX 3000 ; NEOFIX E-117, trade name of Nicca Chemical Co., a polyoxyalkylene polyamine dicyanodiamine, and REDIFLOC 4150, trade name of EKA Chemicals, a polyamine; MADAME  
35 (methacrylatedimethylaminoethyl = dimethylaminoethyl methacrylate)

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or MADQUAT (methacryloxyethyltrimethylammonium chloride) modified polymers, e.g. ROHAGIT KL280, ROHAGIT 210; ROHAGIT SL144, PLEX 4739L, PLEX 3073 from Röhm, DIAFLOC KP155 and other DIAFLOC products from Diafloc Co., and BMB 1305 and other BMB products from EKA  
5 chemicals; cationic epichlorohydrin adducts such as POLYCUP 171 and POLYCUP 172, trade names from Hercules Co.; from Cytec industries : CYPRO products, e.g. CYPRO 514/515/516, SUPERFLOC 507/521/567; cationic acrylic polymers, such as ALCOSTAT 567, trademark of CIBA, cationic cellulose derivatives such as CELQUAT L-200, H-100, SC-  
10 240C, SC-230M, trade names of Starch & Chemical Co., and QUATRISOFT LM200, UCARE polymers JR125, JR400, LR400, JR30M, LR30M and UCARE polymer LK; fixing agents from Chukyo Europe: PALSET JK-512, PALSET JK512L, PALSET JK-182, PALSET JK-220, WSC-173, WSC-173L, PALSET JK-320, PALSET JK-320L and PALSET JK-350; polyethyleneimine and  
15 copolymers, e.g. LUPASOL, trade name of BASF AG; triethanolamine-titanium-chelate, e.g. TYZOR, trade name of Du Pont Co.; copolymers of vinylpyrrolidone such as VIVIPRINT 111, trade name of ISP, a methacrylamido propyl dimethylamine copolymer; with dimethylaminoethylmethacrylate such as COPOLYMER 845 and COPOLYMER  
20 937, trade names of ISP; with vinylimidazole, e.g. LUVIQUAT CARE, LUVITEC 73W, LUVITEC VPI55 K18P, LUVITEC VP155 K72W, LUVIQUAT FC905, LUVIQUAT FC550, LUVIQUAT HM522, and SOKALAN HP56, all trade names of BASF AG; polyamidoamines, e.g. RETAMINOL and NADAVIN, trade marks of Bayer AG; phosphonium compounds such as disclosed in EP 609930 and  
25 other cationic polymers such as NEOFIX RD-5, trademark of Nicca Chemical Co.

The ink receiving layer, and an optional auxiliary layer, such as a backing layer for anti-curl purposes, may further contain well-known  
30 conventional ingredients, such as surfactants serving as coating aids, hardening agents, plasticizers, whitening agents and matting agents.

Surfactants may be incorporated in the layers of the recording element of the present invention. They can be any of the cationic,  
35 anionic, amphoteric, and non-ionic ones as described in JP-A 62-280068 (1987). Examples of the surfactants are N-alkylamino acid

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salts, alkylether carboxylic acid salts, acylated peptides, alkylsulfonic acid salts, alkylbenzene and alkylnaphthalene sulfonic acid salts, sulfosuccinic acid salts,  $\alpha$ -olefin sulfonic acid salts, N-acylsulfonic acid salts, sulfonated oils, alkylsulfonic acid salts, alkylether sulfonic acid salts, alkylallylethersulfonic acid salts, alkylamidesulfonic acid salts, alkylphosphoric acid salts, alkyletherphosphoric acid salts, alkylallyletherphosphoric acid salts, alkyl and alkylallylpolyoxyethylene ethers, alkylallylformaldehyde condensed acid salts, alkylallylethersulfonic acid salts, alkylamidesulfonic acid salts, alkylphosphoric acid salts, alkyletherphosphoric acid salts, alkylallyletherphosphoric acid salts, alkyl and alkylallylpolyoxyethylene ethers, alkylallylformaldehyde condensed polyoxyethylene ethers, blocked polymers having polyoxypropylene, polyoxyethylene polyoxypropylalkylethers, polyoxyethyleneether of glycolesters, polyoxyethyleneether of sorbitanesters, polyoxyethyleneether of sorbitolesters, polyethyleneglycol aliphatic acid esters, glycerol esters, sorbitane esters, propyleneglycol esters, sugaresters, fluoro C<sub>2</sub>-C<sub>10</sub> alkylcarboxylic acids, disodium N-perfluorooctanesulfonyl glutamate, sodium 3-(fluoro-C<sub>6</sub>-C<sub>11</sub>-alkyloxy)-1-C<sub>3</sub>-C<sub>4</sub> alkyl sulfonates, sodium 3-( $\omega$ -fluoro-C<sub>6</sub>-C<sub>8</sub>-alkanoyl-N-ethylamino)-1-propane sulfonates, N-[3-(perfluorooctanesulfonamide)-propyl]-N,N-dimethyl-N-carboxymethylene ammonium betaine, fluoro-C<sub>11</sub>-C<sub>20</sub> alkylcarboxylic acids, perfluoro-C<sub>7</sub>-C<sub>13</sub>-alkyl-carboxylic acids, perfluorooctane sulfonic acid diethanolamide, Li, K and Na perfluoro-C<sub>4</sub>-C<sub>12</sub>-alkyl sulfonates, N-propyl-N-(2-hydroxyethyl)perfluorooctane sulfonamide, perfluoro-C<sub>6</sub>-C<sub>10</sub>-alkylsulfonamide-propyl-sulfonyl-glycinates, bis-(N-perfluorooctylsulfonyl-N-ethanolaminoethyl)phosphonate, mono-perfluoro C<sub>6</sub>-C<sub>16</sub> alkyl-ethyl phosphonates, and perfluoroalkylbetaine.

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Useful cationic surfactants include N-alkyl dimethyl ammonium chloride, palmityl trimethyl ammonium chloride, dodecyldimethylamine, tetradecyldimethylamine, ethoxylated alkyl guanidine-amine complex, oleamine hydroxypropyl bistrimonium chloride, oleyl imidazoline, stearyl imidazoline, cocamine acetate, palmitamine, dihydroxyethylcocamine, cocotrimonium chloride, alkyl polyglycoether ammonium sulphate, ethoxylated oleamine, lauryl pyridinium chloride, N-oleyl-1,3-diaminopropane, stearamidopropyl dimethylamine lactate, coconut fatty amide, oleyl hydroxyethyl imidazoline, isostearyl ethylimidonium ethosulphate, lauramidopropyl PEG-dimoniumchloride phosphate, palmityl trimethylammonium chloride, and cetyltrimethylammonium bromide.

Especially useful are the fluorocarbon surfactants as described in e.g. US-P 4,781,985, having a structure of :

$F(CF_2)_4-9CH_2CH_2SCH_2CH_2N^+R_3X^-$  wherein R is a hydrogen or an alkyl group; and in US-P 5,084,340, having a structure of:

$CF_3(CF_2)_mCH_2CH_2O(CH_2CH_2O)_nR$  wherein  $m = 2$  to  $10$ ;  $n = 1$  to  $18$ ; R is hydrogen or an alkyl group of 1 to 10 carbon atoms. These surfactants are commercially available from DuPont and 3M. The concentration of the surfactant component in the ink-receiving layer is typically in the range of 0.1 to 2 %, preferably in the range of 0.4 to 1.5 % and is most preferably 0.75 % by weight based on the total dry weight of the layer.

The ink-receiving layer and the optional auxiliary layer(s) may be crosslinked to provide such desired features as waterfastness and non-blocking characteristics. The crosslinking is also useful in providing abrasion resistance and resistance to the formation of fingerprints on the element as a result of handling. There are a vast number of known crosslinking agents - also known as hardening agents - that will function to crosslink film forming binders. Hardening agents can be used individually or in combination and in free or in blocked form. A great many hardeners, useful for the present invention, are known, including formaldehyde and free dialdehydes, such as succinaldehyde and glutaraldehyde, blocked

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dialdehydes, active esters, sulfonate esters, active halogen compounds, isocyanate or blocked isocyanates, polyfunctional isocyanates, melamine derivatives, s-triazines and diazines, epoxides, active olefins having two or more active bonds, carbodiimides, zirconium complexes, e.g. BACOTE 20, ZIRMEL 1000 or zirconium acetate, trademarks of MEL Chemicals, titanium complexes, such as TYZOR grades from DuPont, isoxazolium salts substituted in the 3-position, esters of 2-alkoxy-N-carboxy-dihydroquinoline, N-carbamoylpyridinium salts, hardeners of mixed function, such as halogen-substituted aldehyde acids (e.g. mucochloric and mucobromic acids), onium substituted acroleins and vinyl sulfones and polymeric hardeners, such as dialdehyde starches and copoly(acroleinmethacrylic acid), and oxazoline functional polymers, e.g. EPOCROS WS-500, and EPOCROS K-1000 series, and maleic anhydride copolymers, e.g. GANTREZ AN119.

In the practice of this invention boric acid is a preferred crosslinker.

The ink-receiving layer and the optional auxiliary layer(s) may also comprise a plasticizer such as ethylene glycol, diethylene glycol, propylene glycol, polyethylene glycol, glycerol monomethylether, glycerol monochlorohydrin, ethylene carbonate, propylene carbonate, urea phosphate, triphenylphosphate, glycerolmonostearate, propylene glycol monostearate, tetramethylene sulfone, n-methyl-2-pyrrolidone, n-vinyl-2-pyrrolidone.

The different layers can be coated onto the support by any conventional coating technique, such as dip coating, knife coating, extrusion coating, spin coating, slide hopper coating and curtain coating.

The present invention will now be illustrated by the following examples without however being limited thereto.

#### EXAMPLES

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**Example 1**Preparation of the coating solution

5 To apply the light-stabilizing agent to ink jet print media, a coating liquid was prepared by adding 25 parts by solid weight of a 10% aqueous solution of the light-stabilizing compounds ST-1, ST-17, and ST-2 respectively (corresponding to stabilizers 1, 17 and 2 from the compounds list in the Detailed Description section above) to 170  
10 parts of water.

Coating and evaluation of the coated samples

15 The coating solution was applied to a glossy porous medium (Agfajet Universal Instant Dry Photograde Paper Glossy) as the basecoat. In order to change the concentration of the light-stabilizing additive, the thickness of the applied coating solution was varied. The application was performed by means of a doctor blade coater. The comparative samples were obtained by simply applying the aqueous  
20 solution to the recording media without the stabilizing agent. After the media were allowed to dry at room temperature for 24 hours, color patches with 50% and 100% ink of cyan, magenta, yellow and black were printed by means of a printer HP970Cxi (trademark of Hewlett-Packard). The black patches were obtained by printing cyan,  
25 magenta and yellow. The light-fastness was evaluated by measuring the relative optical density loss of the printed samples after being exposed to light in a fade-o-meter, XENOTEST 150 (trademark: Original Hanau) with 180 kLux during 16 hours. The results are summarised in table 1.

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TABLE 1 : comparison of optical density loss of printed media (50% patches) treated with and without light-stabilizing additive:

Color	Relative optical density loss (%)			
	Without ST	With 1.0 g/m <sup>2</sup> of ST-1	With 1.0 g/m <sup>2</sup> of ST-17	With 0.5 g/m <sup>2</sup> of ST-2
Yellow	30.8	23.6	13.5	21.8
Magenta	41.9	19.4	13.6	20.9
Cyan	41.2	32.1	28.9	33.3
Black	36.2	18.6	15.5	20.0

As can be seen from the table, the porous materials impregnated with the stabilizers ST-1, ST-17, and ST-2 show an important improvement in light-fastness.

### Example 2

Ink jet recording media (invention and comparison) were prepared by coating on a resin-coated paper an ink receiving layer which composition is shown in table 2. The coating weight of the inorganic pigment was 30.0 g/m<sup>2</sup>. On top of this layer, a gloss improving layer is coated containing 100 parts of the commercial boehmite DISPERAL HP 14/2, from Sasol Co., a 25 % dispersion in water, 2 parts of a polyvinyl alcohol GOHSEFIMER K210 from Nippon Goshei Co., 0.2 parts of boric acid, and 0.8 parts of cetyltrimethylammonium bromide. The coating thickness of the top layer was chosen to achieve a pigment coating weight of 5 g/m<sup>2</sup>. The invention samples contained 20 parts of the stabilizing agents ST-1 and ST-2 respectively in the top layer, the comparative sample did not.

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TABLE 2

Compositions	
Alumina (Cab-o-Sperse PG003: 40%, supplied by Cabot Corp.)	704.3 g
Polyvinyl alcohol (Gohsefimer K210, supplied by Nippon Gohsei)	18.3 g
Pseudo-boehmite DISPERAL P3 (trade name of Sasol Co.)	28.2 g
Boric acid	1.8 g
Deionised water	477.5 g

- 5 Color patches with 50% and 100% ink of cyan, magenta, yellow and black were printed on both samples by means of a printer HP970Cxi (trademark of Hewlett-Packard). The light-fastness was evaluated by measuring the relative optical density loss of the printed samples after being exposed to light in a fade-o-meter, XENOTEST 150
- 10 (trademark of Original Hanau) with 180 kLux during 16 hours. The relative loss (%) in density of the four 50% color patches together, due to the light fading are shown in table 3.

TABLE 3

15

Color	Comp. Sample without ST	Inv. sample with ST-1	Inv. sample with ST-2
Yellow	42.1	39.6	37.5
Magenta	80.3	59.7	56.3
Cyan	49.0	44.2	43.4
Black	54.4	41.4	45.0

As can be seen, the color stability, especially for the magenta ink and the black color (obtained from cyan, magenta and yellow ink),



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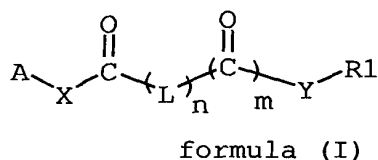
was much improved when compounds ST-1 and ST-2 were introduced in the top layer.

- 5 Having described in detail preferred embodiments of the current invention, it will now be apparent to those skilled in the art that numerous modifications can be made therein without departing from the scope of the invention as defined in the appending claims.

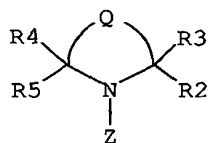


**[CLAIMS]**

1. An ink jet recording material comprising a support and at least one ink receiving layer containing a binder and a compound according to following general formula (I) :



wherein A is represented by following formula :



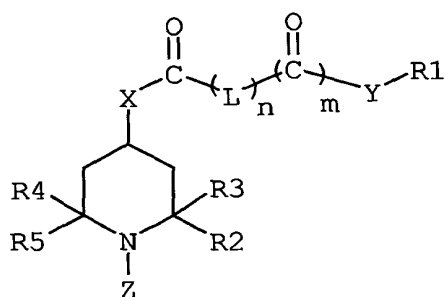
- wherein,
- Q represents the necessary atoms to complete a five- or six-membered ring;
- R2 to R5 independently represent a substituted or unsubstituted C1 to C6 aliphatic group;
- Z is selected from the group consisting of hydrogen, a substituted or unsubstituted aliphatic group, an acyl group, an oxy radical, a hydroxyl group, an alkoxy group and an acyloxy group;
- L represents a divalent linking group;
- X and Y are independently selected from an oxygen and NR6, wherein R6 is selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group; X is linked to A via one of the atoms of Q;

- 30 -

R1 represents a non-aromatic moiety comprising at least one hydroxyl group;

n and m independently represent 1 or 0.

2. An ink jet material according to claim 1 wherein said compound is represented by following general formula (II) :



formula (II)

wherein,

R2 to R5 independently represent a substituted or unsubstituted C1 to C6 aliphatic group;

Z is selected from the group consisting of hydrogen, a substituted or unsubstituted aliphatic group, an acyl group, an oxy radical, a hydroxyl group, an alkoxy group and an acyloxy group;

L represents a divalent linking group;

X and Y are independently selected from an oxygen and NR6,

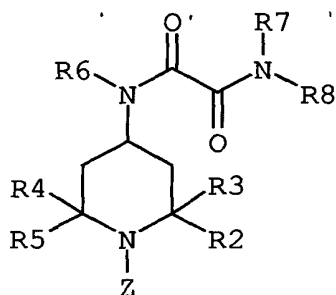
wherein R6 is selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group;

R1 represents a non-aromatic moiety comprising at least one hydroxyl group;

n and m independently represent 1 or 0.

3. An ink jet recording material according to claim 2 wherein said compound is represented by following general formula (III) :

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formula (III)

5 wherein,

R2 to R5 independently represent a substituted or unsubstituted C1 to C6 aliphatic group;

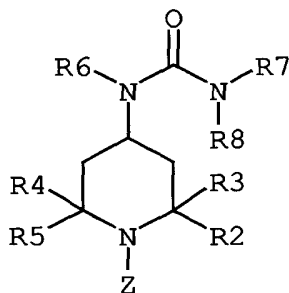
10 Z is selected from the group consisting of hydrogen, a substituted or unsubstituted aliphatic group, an acyl group, an oxy radical, a hydroxyl group, an alkoxy group and an acyloxy group;

15 R6 is selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group;

20 R7 and R8 are independently selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group, with the proviso that at least one of R7 and R8 represents a non-aromatic moiety comprising at least one hydroxyl group.

4. An ink jet recording material according to claim 2 wherein said compound is represented by following general formula (IV) :

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formula (IV)

wherein,

5 R2 to R5 independently represent a substituted or unsubstituted C1 to C6 aliphatic group;

Z is selected from the group consisting of hydrogen, a substituted or unsubstituted aliphatic group, an acyl group, an oxy radical, a hydroxyl group, an alkoxy group and an acyloxy group;

10 R6 is selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group;

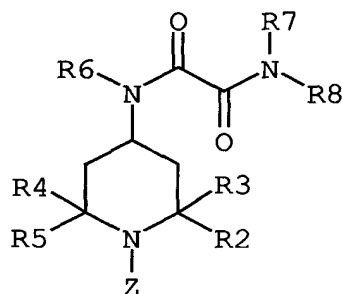
15 R7 and R8 are independently selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group, with the proviso that at least one of R7 and R8 represents a non-aromatic moiety comprising at least one hydroxyl group.

20 5. An ink jet recording material according to any of claims 1 to 4 wherein said recording material further comprises a pigment in at least one ink receiving layer.

25 6. An ink jet recording material according to claim 5 wherein said pigment is an inorganic pigment.

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7. An ink jet recording material according to claim 6 wherein said inorganic pigment is chosen from the group consisting of silica, alumina, alumina hydrate, and aluminum trihydroxide.
8. An ink jet recording material according to any of claims 1 to 7 wherein wherein said binder is a polyvinyl alcohol.
9. An ink jet recording material according to any of claims 1 to 8 wherein the ink receiving layer is a double layer and the compound according to general formula (I) is incorporated in the upper ink receiving layer.
10. A compound according to following formula :



wherein,

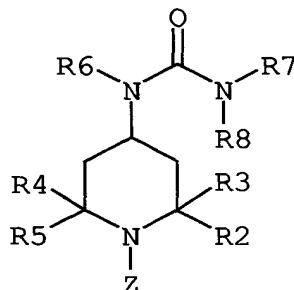
- R2 to R5 independently represent a substituted or unsubstituted C1 to C6 aliphatic group;
- Z is selected from the group consisting of hydrogen, a substituted or unsubstituted aliphatic group, an acyl group, an oxy radical, a hydroxyl group, an alkoxy group and an acyloxy group;
- R6 is selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group;
- R7 and R8 are independently selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted

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aromatic group, and a substituted or unsubstituted heteroaromatic group, with the proviso that at least one of R7 and R8 represents a non-aromatic moiety comprising at least two hydroxyl groups.

11. A compound according to following formula :

5



wherein,

10 R2 to R5 independently represent a substituted or unsubstituted C1 to C6 aliphatic group;

Z is selected from the group consisting of hydrogen, a substituted or unsubstituted aliphatic group, an acyl group, an oxy radical, a hydroxyl group, an alkoxy group and an acyloxy group;

15 R6 is selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group;

20 R7 and R8 are independently selected from the group consisting of hydrogen, a substituted or unsubstituted, saturated or unsaturated aliphatic group, a substituted or unsubstituted aromatic group, and a substituted or unsubstituted heteroaromatic group, with the proviso that at least one of R7 and R8 represents a non-aromatic moiety comprising at least one hydroxyl group.

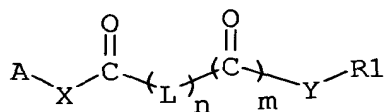


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**[ABSTRACT]****IMPROVED INK JET RECORDING MATERIAL**

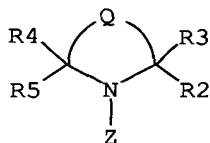
5

An ink jet recording material is disclosed comprising a support and at least one ink receiving layer containing a binder and a light-stabilizing compound according to following general formula (I) :



10

wherein A is represented by following formula :



the symbols of which are defined in the claims and description.

15 The finished ink jet image shows an improved light-fastness.

